INSTITUTE OF PAPER CHEMISTRY
Appleton · Wisconsin

THE INTERACTION OF MICROWAVE RADIATION WITH PAPER–WATER SYSTEMS

Project 3322

Report Two

A Progress Report
to
MEMBERS OF THE INSTITUTE OF PAPER CHEMISTRY

October 9, 1979

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Appleton, Wisconsin

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SUMMARY

Progress Report 1 discussed the effect of process variables on the dielectric constant of water-paper systems at "dry end" moisture levels. This report describes additional work at higher levels. The experimental procedure differs from the "dry end" tests in that the samples must be sealed in plastic "bags" to maintain their high moisture contents. The major observations made are: (1) At high moisture levels, the water dominates the complex dielectric constant. The effects of wet pressing, refining, and calendering are qualitatively the same as observed at the "dry end," but of less relative magnitude. (2) There is evidence that the strength of the bonding of the water to the fiber depends on the furnish. (3) The in-plane anisotropy of the dielectric constant is still present at high moisture levels.

INTRODUCTION

This report presents the results of our measurements of the dielectric constant of water-paper systems at high moisture levels at microwave frequencies. An earlier report, Progress Report 1* (PR1) was concerned with samples having moisture levels under 15%. PR1 provides information about the effect of furnish and paper machine variables on the dielectric constant of paper. The parameters investigated were wet pressing, refining, calendering, basis weight, TiO_2 content, clay content, sheet orientation, temperature, and frequency. The complex dielectric constant is a fundamental parameter, and knowledge of the dependence of this parameter on process variables is necessary for understanding the limitations of microwave moisture gages and designing gages less sensitive to process changes.

At the wet end of the paper machine moisture levels considerably greater than 15% are encountered. Since microwave moisture gages are used at the wet end, we felt it was necessary to extend the project to investigate the effect of certain parameters at high moistures. The results of this study are reported here.

PR1 describes in detail the experimental apparatus developed to make accurate dielectric constant measurements on thin samples. The experimental procedure used here is essentially the same, except the sample preparation procedure is different at the higher moisture levels. These samples must be sealed in plastic bags in order to maintain constant moisture content during the test. Procedural details will be discussed in this report only when they differ from those in PR1.

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*Project 3322, The Interaction of Microwave Radiation with Paper-water Systems. Report One. October 13, 1978.

EXPERIMENTAL PROCEDURES

The moisture content of the samples prepared for the "dry end" measurements were varied by adjusting the relative humidity of the laboratory. The data presented in this report is taken on wetter samples. The samples are maintained at high moisture content by sealing them in 0.0005-inch Saran* wrap. Preliminary experiments conducted on the samples at 50% R.H. showed that the effect of the Saran wrap on the measured microwave dielectric constants was negligible (no effects were detected).

The samples used in this investigation are described in Table I. They were chosen from the paper grades tested in the "dry end" study. One phenomenon observed in the previous study was the in-plane anisotropy in the dielectric constant of machine-made papers. To see if this persisted at high moisture levels, a machine-made newsprint, NM052, was tested with samples cut to align the machine direction and the electric field and to align the cross direction and the electric field. Of primary interest in the high moisture studies were the effects of wet pressing and refining. Unbleached kraft (UK) handsheet samples, with varying degrees of refining and wet pressing, were used. Calendered and uncalendered, bleached kraft handsheets were also studied. All readings were made at a temperature of 23°C and a frequency of 9.6 GHz.

The details of sample preparation and measurement are enumerated below:

(1) The samples were first preconditioned at 15% R.H., 23°C, and then at 50% R.H., 23°C. A series of rectangular specimens was cut from

*Saran. Dow Chemical Company.

each paper sample. The size of the rectangle was such that the "unbagged" sample would fill the waveguide opening in the window shim sample holder (see PR1 for a description of the window shims).

(2) The specimens were weighed and the caliper was measured. For each sample, three rectangular specimens were selected so that the weights were equal to within ± 0.0001 mg and the calipers equal to within ± 0.0001 inch.

(3) The specimens were soaked in distilled water for an hour, removed, and allowed to drain. Depending on the caliper, one, two, or three sheets were sealed together for testing at the highest moisture content. This was done by using a piece of 0.0005 inch Saran wrap three times longer than the height of the rectangles and slightly wider than the width of the rectangles. The samples were stacked and wrapped so that two layers of Saran were on one side of the stack and one layer was on the other side. The edges were sealed by placing the stack between two pieces of metal, flush with the edge of the specimens, and melting the excess plastic with a match.

(4) The sealed specimens were weighed and the caliper was measured.

(5) The sealed specimens were carefully placed in a window shim. The assembly was inserted in the waveguide and the standing wave patterns for specific short locations were determined as described in PR1.

(6) The sealed specimens were removed from the waveguide and immediately reweighed to insure that no moisture had been lost.

(7) The seal was clipped at one edge and the paper stack was removed. The plastic was weighed and the specimen was recorded as the sealed weight less the weight of the Saran. The caliper was recorded as the measured caliper of the sealed sample less 0.0015 inch.

(8) The raw data was inputed to the computer and the program described in PR1 was used to calculate the complex dielectric constant and estimate the uncertainty.

(9) The specimens were allowed to dry to a new moisture content and were then resealed and the measurements repeated.

TABLE I

SAMPLE CHARACTERISTICS

Sample No.	Description	Oven-dry Basis Wt., g/m^2	Wet Pressing Pressure, psi	Other
NM052	Machine-made newsprint	44.3		E. field parallel to M.D.
NM052	Machine-made newsprint	44.7		E. field parallel to C.D.
UK205-1	Unbleached kraft	198.4	10	Refining time, 5 min
UK205-2	Unbleached kraft	199.2	50	Refining time, 5 min
UK205-3	Unbleached kraft	199.6	84	Refining time, 5 min
UK205-4	Unbleached kraft	200.0	400	Refining time, 5 min
UK205-5	Unbleached kraft	201.3	50	Refining time, 25 min
BK060-00	Bleached kraft	58.6	50	
BK060-02	Bleached kraft	58.6	50	Three calender passes

As the specimens become progressively dryer, in order to achieve sufficient instrument sensitivity, it was sometimes necessary to place additional sheets in the specimen stack. The total number of sheets in the stack was always three or less. To insure that the new sheets were identical to the others, they had simultaneously been treated along with the original sheets, but sealed in separate containers.

RESULTS AND DISCUSSION

Table II provides a complete listing of the results. Some data from the "dry end" study are also included for the paper grades studied at the higher moistures. It should be noted, however, that the "dry end" samples are different specimens than those used in this study and the specimens were not sealed in plastic.

The effect of wet pressing and refining on the dielectric constant of unbleached kraft handsheets is demonstrated in Fig. 1 and 2, where the real and imaginary parts of the dielectric constant, respectively, are plotted as a function of gravimetric moisture content. At constant moisture content it is clear that the more heavily treated samples have the higher dielectric constants. This is largely due to the increase in apparent density with refining and wet pressing. At high moistures the water in the water-paper system is the major contributer to the system's interaction with microwave radiation. Since the denser sheets have more moisture per unit volume, they have a higher dielectric constant at the same gravimetric moisture content.

The three major factors that determine the magnitude of the apparent dielectric constant of wet paper sheets are: (1) the density of the moisture; (2) the degree to which molecular bonding between the water and the fiber inhibits the ability of the water to interact with the microwave radiation; and (3) the geometric distribution of the water at the fiber level. In order to investigate the effects of refining and wet pressing on factors 2 and 3, dielectric constants should be compared at constant water density. This is done in Fig. 3 and 4 for the UK samples by plotting the log of the dielectric constant against the log of the water density. Notice that at high water densities the

TABLE II

SUMMARY OF RESULTS,
23°C, 9.6 GHz

Sample: NM052
Orientation:
ODBW: 44.27 g/m² per sheet

Water Basis Wt./Sht., g/m²	Grav. Moist., %	P_{H_2O}, g/cm³	No. Sheets	Caliper per Sheet, inch	ε'	ε"
117.43	72.62	0.811	2	0.0057	32.14	8.77
108.15	70.95	0.760	2	0.0056	27.83	7.50
95.93	68.42	0.687	2	0.0055	26.30	6.72
84.54	65.63	0.616	2	0.0054	23.60	6.74
75.05	62.90	0.547	2	0.0054	22.29	5.97
63.04	58.74	0.477	2	0.0052	16.17	4.60
53.97	54.94	0.409	2	0.0052	14.25	4.17
51.02	53.54	0.387	3	0.0052	13.48	4.40
44.84	50.31	0.347	3	0.0051	11.71	3.69
36.40	45.12	0.286	3	0.0050	9.70	3.33
28.39	39.07	0.229	3	0.0049	7.60	2.63
19.82	30.92	0.164	3	0.0048	5.43	1.87
12.51	22.03	0.106	3	0.0047	3.75	1.06
6.61	12.98	0.058	3	0.0046	2.49	0.31
*4.43	9.03	0.043	6	0.0040	2.11	0.19

Sample: NM052
Orientation:
ODBW: 44.70 g/m² per sheet

Water Basis Wt./Sht., g/m²	Grav. Moist., %	P_{H_2O}, g/cm³	No. Sheets	Caliper per Sheet, inch	ε'	ε"
86.65	65.97	0.632	2	0.0054	19.53	5.38
68.94	60.67	0.503	2	0.0054	16.71	5.23
56.71	55.93	0.421	2	0.0053	15.03	4.30
44.70	50.00	0.345	2	0.0051	11.02	2.14
34.86	43.82	0.274	3	0.0050	7.79	2.59
27.55	38.13	0.221	3	0.0049	6.66	2.17
19.68	30.57	0.161	3	0.0048	4.86	1.41
13.77	23.56	0.115	3	0.0047	3.56	0.91
8.57	16.09	0.073	3	0.0046	2.71	0.36
5.20	10.42	0.045	3	0.0045	2.06	0.08
*4.50	9.13	0.044	6	0.0040	1.99	0.15

Sample: BK060-00
Orientation:
ODBW: 58.57 g/m² per sheet

Water Basis Wt./Sht., g/m²	Grav. Moist., %	P_{H_2O}, g/cm³	No. Sheets	Caliper per Sheet, inch	ε'	ε"
78.00	57.11	0.610	3	0.0050	30.26	10.27
59.27	50.30	0.470	3	0.0050	18.78	6.82
46.69	44.36	0.380	3	0.0048	13.89	4.56
32.29	35.54	0.265	3	0.0048	10.46	3.71
16.22	21.68	0.139	3	0.0046	5.10	1.72
5.45	8.52	0.058	3	0.0037	2.84	0.32
*8.87	13.02	0.093	6	0.0038	3.45	1.02
*4.50	7.06	0.049	6	0.0036	2.66	0.31
*2.28	3.71	0.025	6	0.0036	2.29	0.14

Sample: BK060-02
Orientation:
ODBW: 58.57 g/m² per sheet

Water Basis Wt./Sht., g/m²	Grav. Moist., %	P_{H_2O}, g/cm³	No. Sheets	Caliper per Sheet, inch	ε'	ε"
82.62	58.51	0.707	3	0.0046	33.50	10.64
73.81	55.76	0.646	3	0.0045	29.04	9.72
65.14	52.66	0.583	3	0.0044	25.45	7.13
54.10	48.01	0.488	3	0.0044	23.24	6.84
41.80	41.64	0.380	3	0.0043	14.54	4.22
27.68	32.09	0.253	3	0.0043	10.44	3.47
14.40	19.73	0.153	3	0.0037	5.88	1.69
5.73	8.91	0.071	3	0.0032	3.05	0.24
*8.36	12.37	0.106	6	0.0031	3.64	0.85
*4.50	7.06	0.064	6	0.0028	2.99	0.31
*2.41	3.91	0.034	6	0.0028	2.57	0.14

Sample: UK205-1
Orientation:
ODBW: 198.36 g/m² per sheet

Water Basis Wt./Sht., g/m²	Grav. Moist., %	P_{H_2O}, g/cm³	No. Sheets	Caliper per Sheet, inch	ε'	ε"
381.92	65.87	0.569	1	0.0265	21.37	8.92
340.09	63.22	0.508	1	0.0264	18.96	7.55
308.30	60.91	0.468	1	0.0260	16.74	6.36
262.28	57.00	0.400	1	0.0259	15.00	5.84
207.48	51.19	0.324	1	0.0253	10.70	4.05
171.51	46.43	0.271	1	0.0250	8.59	3.26
125.08	38.73	0.203	1	0.0243	6.98	2.55
84.08	29.82	0.143	1	0.0232	4.97	1.72
47.27	19.28	0.092	1	0.0203	3.13	0.83
23.43	10.59	0.048	1	0.0194	2.11	0.19
*30.78	13.36	0.062	2	0.0196	2.46	0.52
*17.48	8.05	0.037	2	0.0188	1.98	0.18
*9.50	4.54	0.020	2	0.0182	1.80	0.07

Sample: UK205-2
Orientation:
ODBW: 199.20 g/m² per sheet

Water Basis Wt./Sht., g/m²	Grav. Moist., %	P_{H_2O}, g/cm³	No. Sheets	Caliper per Sheet, inch	ε'	ε"
492.35	71.25	0.793	1	0.0245	33.80	15.60
449.69	69.35	0.740	1	0.0240	30.93	13.26
408.27	67.26	0.683	1	0.0236	26.47	10.00
340.92	63.18	0.573	1	0.0235	19.94	7.47
280.27	58.52	0.477	1	0.0232	16.72	6.20
235.09	54.19	0.403	1	0.0230	13.38	4.92
192.01	49.14	0.337	1	0.0225	10.51	4.37
156.87	44.12	0.281	1	0.0220	8.91	3.19
109.18	35.46	0.202	1	0.0213	6.11	2.43
73.62	27.04	0.148	1	0.0197	5.00	1.84
23.43	10.55	0.054	1	0.0172	2.26	0.22
*30.59	13.27	0.070	2	0.0173	2.62	0.59
*17.48	8.04	0.042	2	0.0166	2.15	0.23
*9.31	4.45	0.023	2	0.0161	1.89	0.07

Sample: UK205-3
Orientation:
ODBW: 199.62 g/m² per sheet

Water Basis Wt./Sht., g/m²	Grav. Moist., %	P_{H_2O}, g/cm³	No. Sheets	Caliper per Sheet, inch	ε'	ε"
335.49	62.75	0.607	1	0.0218	26.15	9.82
287.38	59.07	0.525	1	0.0216	20.60	7.51
240.53	54.71	0.444	1	0.0214	16.48	6.18
197.02	49.74	0.370	1	0.0210	12.84	5.36
143.90	41.95	0.276	1	0.0206	9.12	3.80
107.09	34.97	0.209	1	0.0202	7.13	2.89
61.91	23.72	0.130	1	0.0188	4.89	1.66
23.43	10.53	0.057	1	0.0163	2.45	0.25
*30.21	13.11	0.076	2	0.0157	2.92	0.70
*16.91	7.79	0.044	2	0.0150	2.24	0.23
*8.74	4.18	0.024	2	0.0145	2.00	0.09

Sample: UK205-4
Orientation:
ODBW: 200.04 g/m² per sheet

Water Basis Wt./Sht., g/m²	Grav. Moist., %	P_{H_2O}, g/cm³	No. Sheets	Caliper per Sheet, inch	ε'	ε"
407.85	67.15	0.732	1	0.0220	31.94	13.01
357.66	64.19	0.648	1	0.0218	28.66	9.49
314.99	61.22	0.578	1	0.0215	22.76	7.78
266.05	57.14	0.500	1	0.0210	19.51	6.92
221.71	52.63	0.427	1	0.0205	15.84	5.72
185.73	48.21	0.368	1	0.0199	12.80	5.01
144.74	42.04	0.298	1	0.0192	10.30	4.20
106.67	34.84	0.226	1	0.0186	8.41	3.08
68.18	25.47	0.151	1	0.0178	5.55	1.84
20.50	9.32	0.055	1	0.0147	2.46	0.20
*30.79	13.32	0.092	2	0.0132	3.45	0.91
*17.29	7.95	0.054	2	0.0126	2.56	0.29
*9.12	4.36	0.029	2	0.0122	2.25	0.11

Sample: UK205-5
Orientation:
ODBW: 201.30 g/m² per sheet

Water Basis Wt./Sht., g/m²	Grav. Moist., %	P_{H_2O}, g/cm³	No. Sheets	Caliper per Sheet, inch	ε'	ε"
381.08	65.49	0.671	1	0.0224	26.58	12.63
342.18	63.02	0.606	1	0.0223	25.60	9.39
299.09	59.83	0.537	1	0.0220	22.24	8.30
262.70	56.68	0.480	1	0.0216	19.12	7.07
209.57	51.07	0.398	1	0.0208	16.13	5.89
156.45	43.79	0.306	1	0.0202	11.27	4.08
113.78	36.17	0.230	1	0.0195	8.24	3.13
78.22	28.04	0.166	1	0.0186	6.50	2.47
46.85	18.92	0.112	1	0.0165	4.48	1.31
20.50	9.26	0.053	1	0.0154	2.42	0.21
*30.40	13.14	0.084	2	0.0143	3.21	0.82
*16.72	7.68	0.049	2	0.0135	2.43	0.26
*8.55	4.08	0.026	2	0.0131	2.14	0.09

*Dry end data taken from Table I PR1.

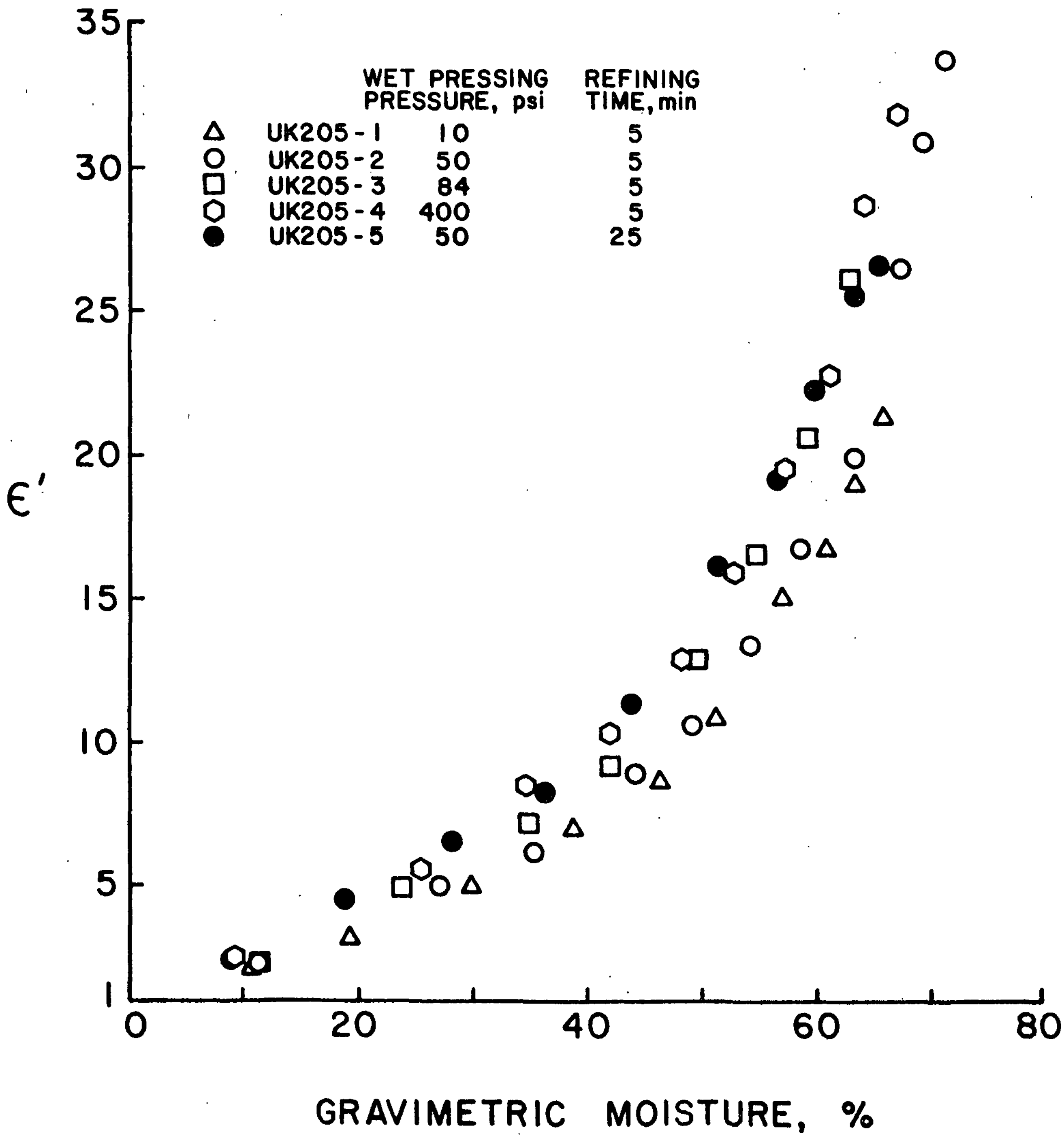

Figure 1. The Real Part of the Dielectric Constant as a Function of Percent Moisture for Unbleached Kraft Samples Differing in Wet Pressing and Refining Treatments. Data Obtained at a Frequency of 9.6 GHz

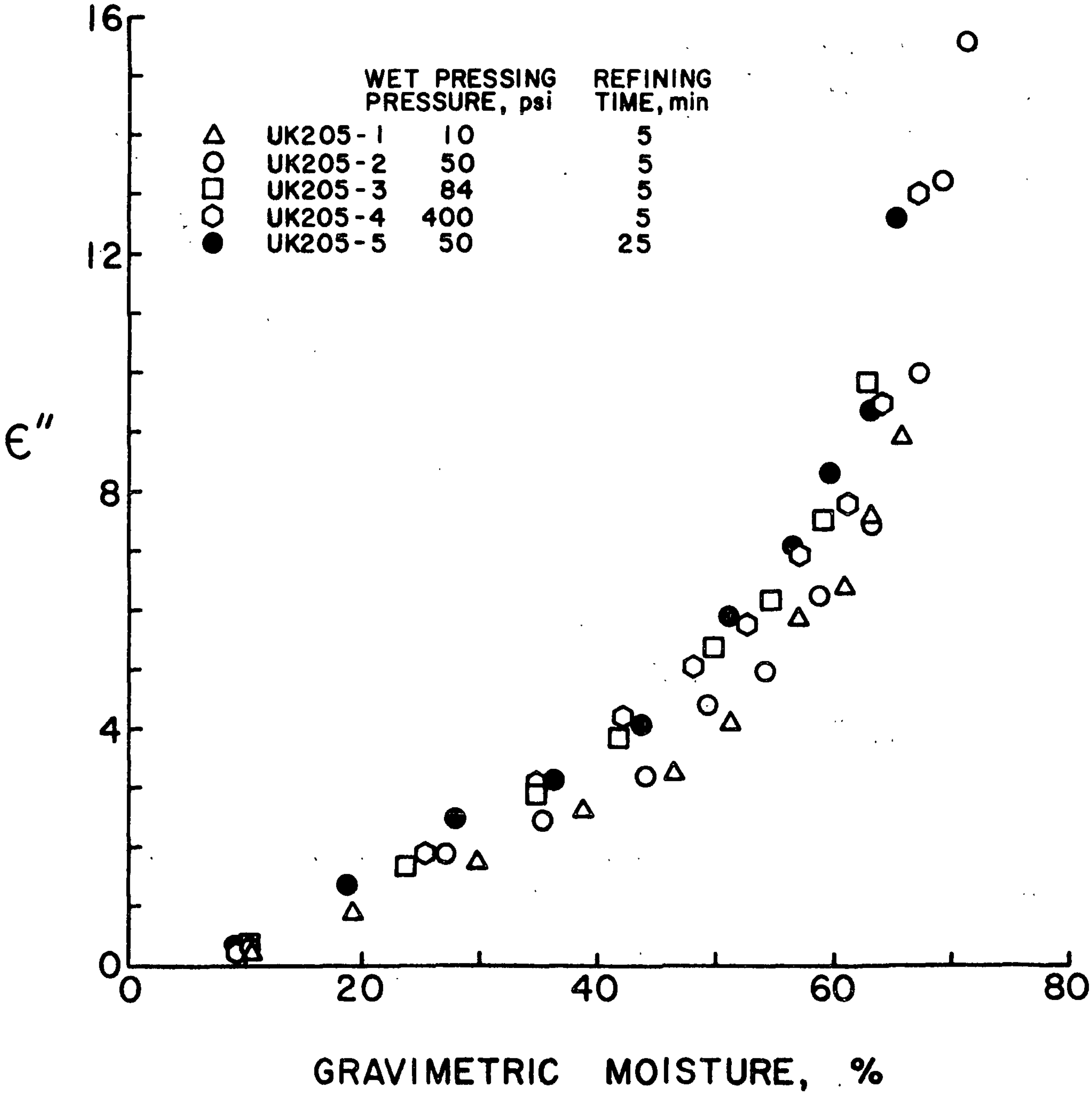

Figure 2. The Imaginary Part of the Dielectric Constant as a Function of Percent Moisture for Unbleached Kraft Samples Differing in Wet Pressing and Refining Treatments. Data Obtained at a Frequency of 9.6 GHz

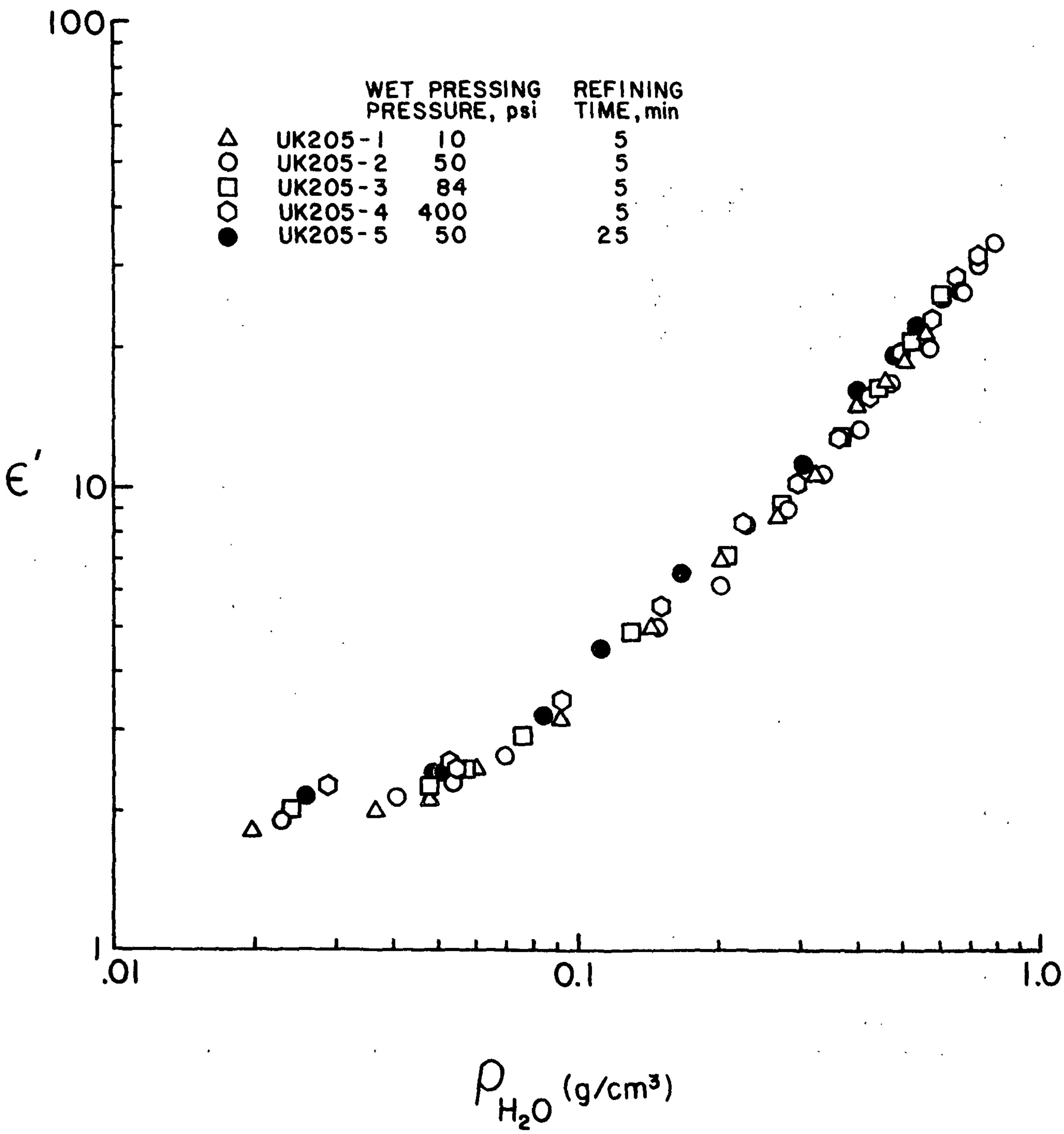

Figure 3. The Data of Figure 1 Replotted as log ε' *versus* log ρH_2O. ρH_2O is the Weight of Water Divided by the Sample Volume. Measurements at 9.6 GHz

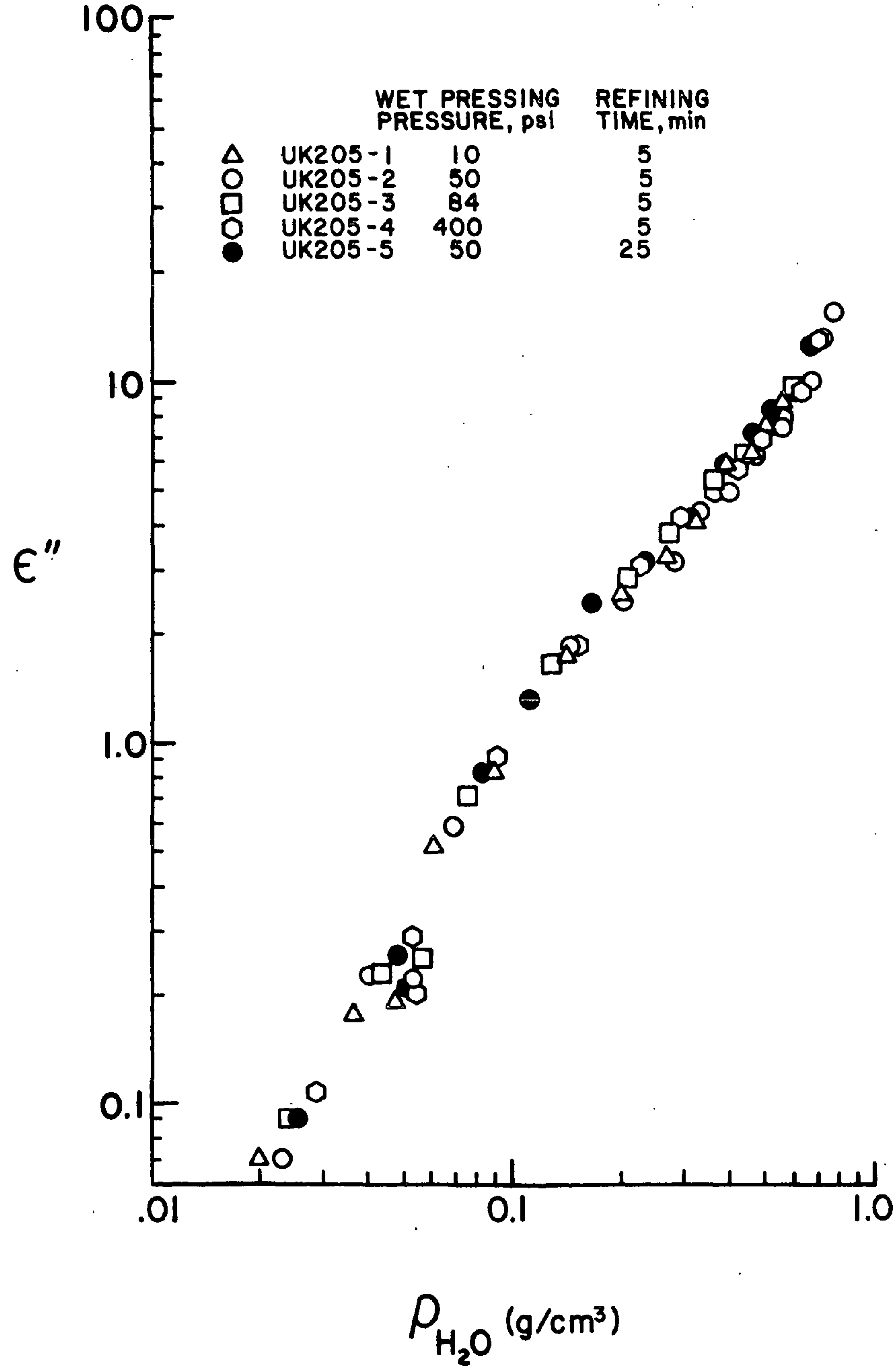

Figure 4. The Data of Figure 2 Replotted as log ε" _versus_ log ρH_2O. Measurements at 9.6 GHz

more highly treated (refined or wet pressed) samples have only a slightly greater dielectric constant. Data taken at the "dry end" are included at the bottom left section of the graph. In this region the fiber makes a significant contribution to the real part of the dielectric constant, and differences in ε' are more pronounced. The fiber does not contribute significantly to ε'' (Fig. 4), i.e., the fiber is not a "lossy" material.

The role of the second factor at high moistures can be estimated by extrapolating the curves to a water density of one and comparing this with the dielectric constant of pure water. The third factor is probably not important in this case because the geometry of the water at the fiber level must become highly connected as ρH_2O approaches one. The result of this extrapolation for the unbleached kraft handsheets is $\varepsilon \sim 45 + 20i$, while the dielectric constant of water is 55 + 30i at this frequency and temperature.

The effect of calendering on the dielectric constant of a bleached kraft handsheet is shown in Fig. 5 and 6. As in the case of refining and wet pressing, the more heavily treated samples have a greater apparent density and thereby a higher dielectric constant at the same moisture content. Figures 7 and 8 are log-log plots of dielectric constant against water density. It was stated in PR1 that apparent density increases, due to calendering, were less effective in increasing the dielectric constant than were increases due to refining or wet pressing. This phenomenon is not as pronounced at high moistures, but it is observed by comparing Fig. 3 and 4 with Fig. 7 and 8, respectively. The effect of treatment on the UK samples was to increase slightly the dielectric constant (both ε' and ε'') at the same water density; for the BK samples the ε' of the more calendered sheet was unchanged and the ε'' decreased with treatment.

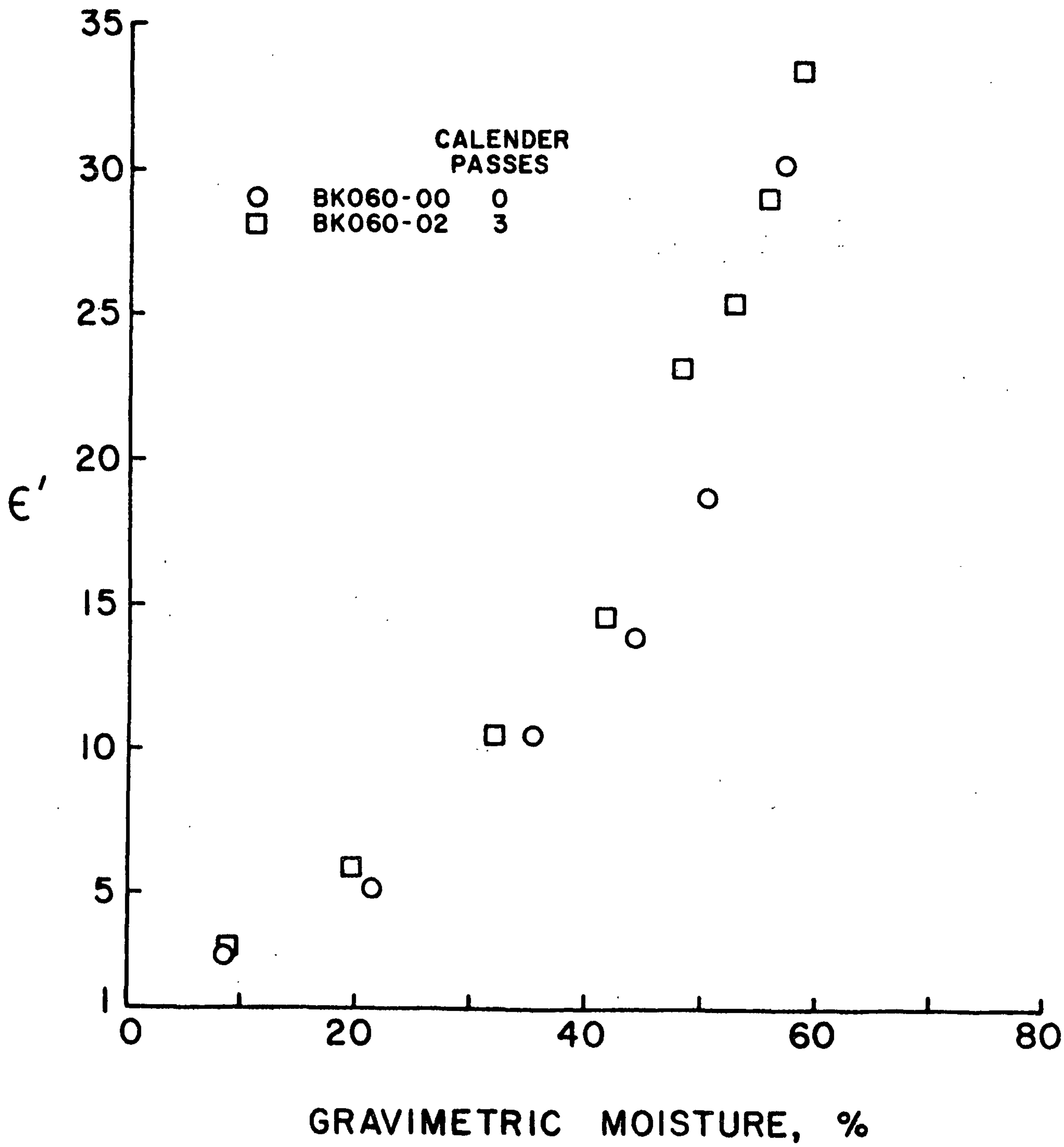

Figure 5. The Effect of Calendering on ε' as a Function of Percent Moisture. Measurements at 9.6 GHz

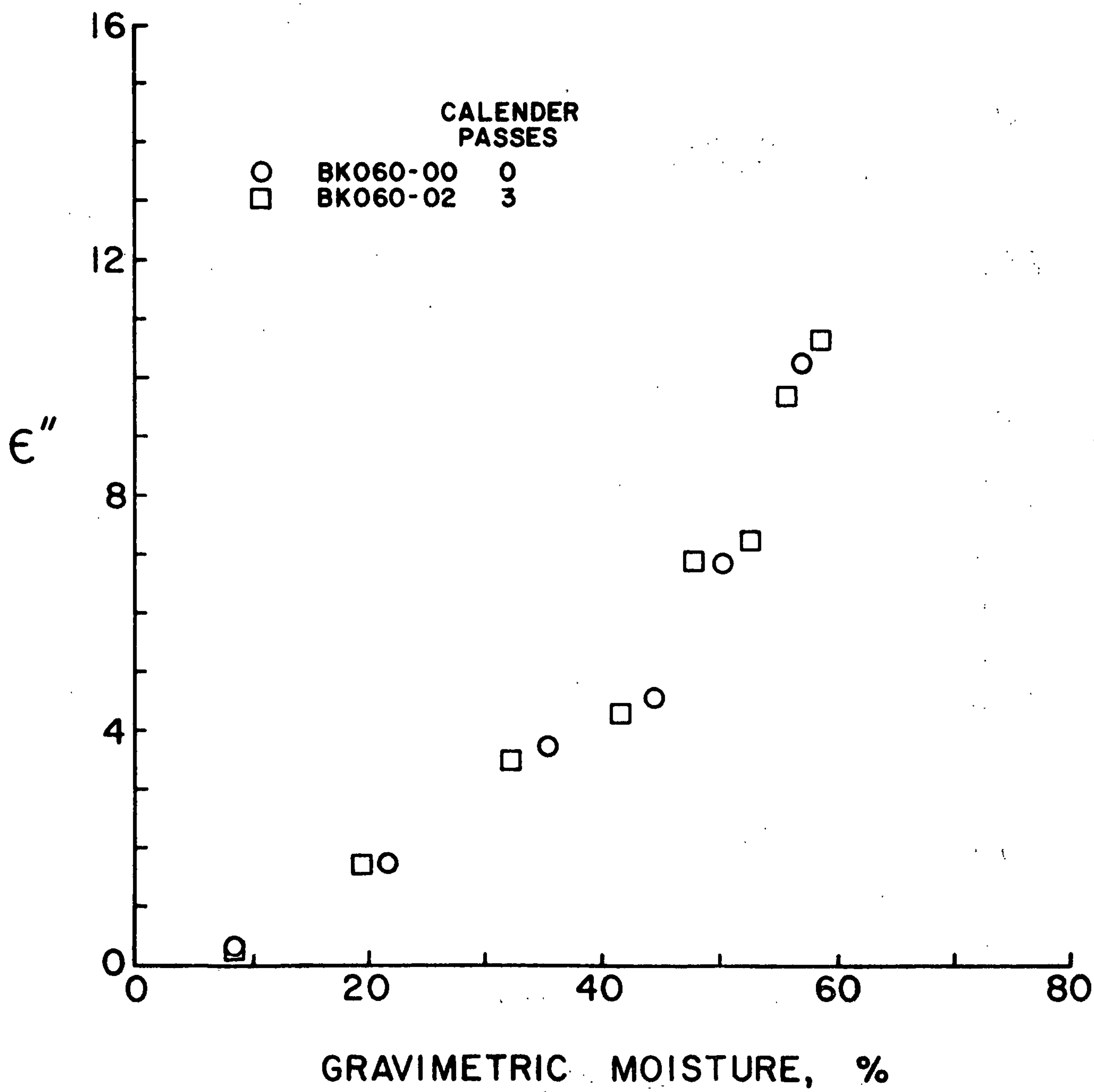

Figure 6. The Effect of Calendering on ε" as a Function of Percent Moisture. Measurements at 9.6 GHz

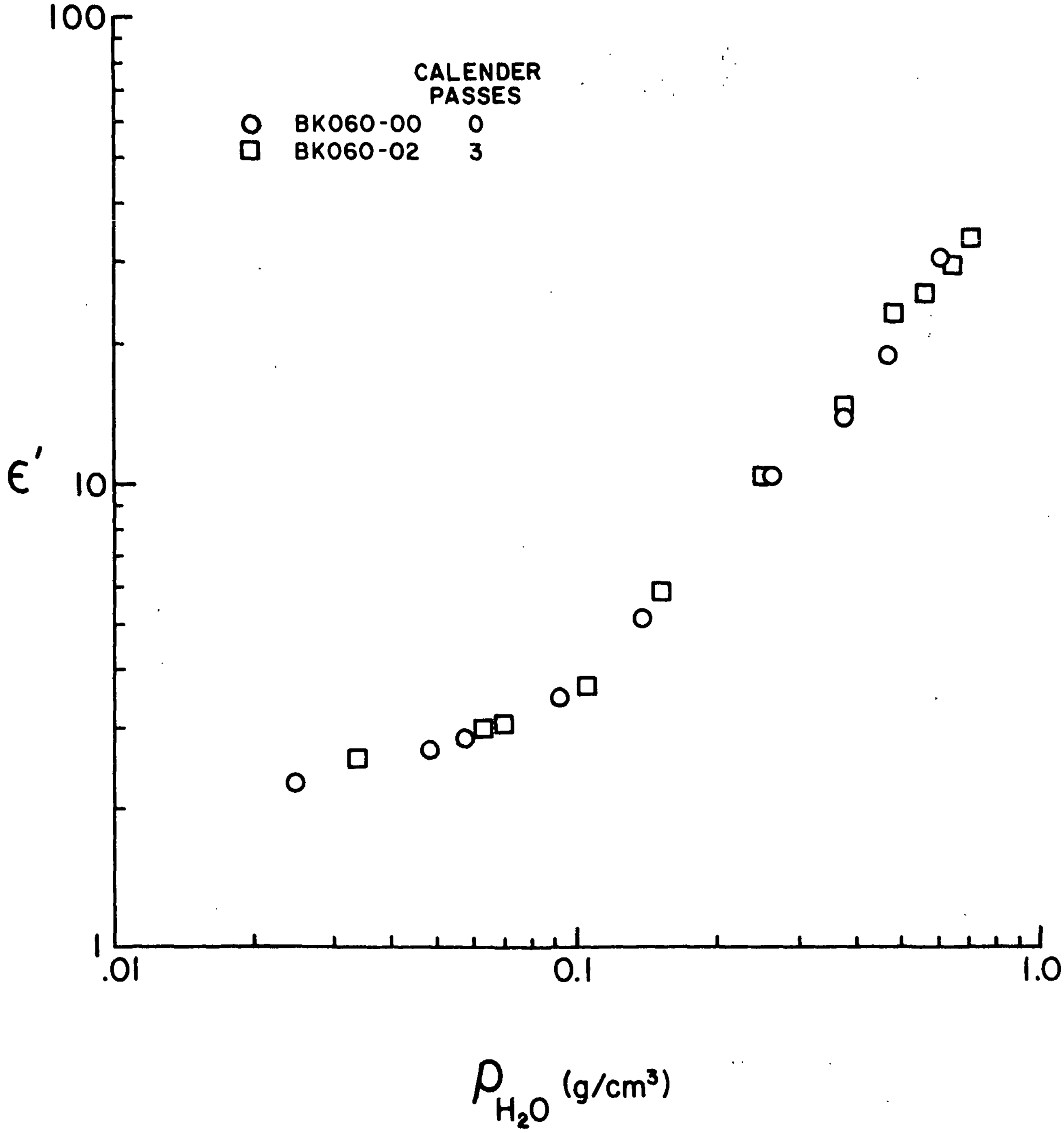

Figure 7. The Data of Figure 5 Replotted as log ε' versus log ρH_2O

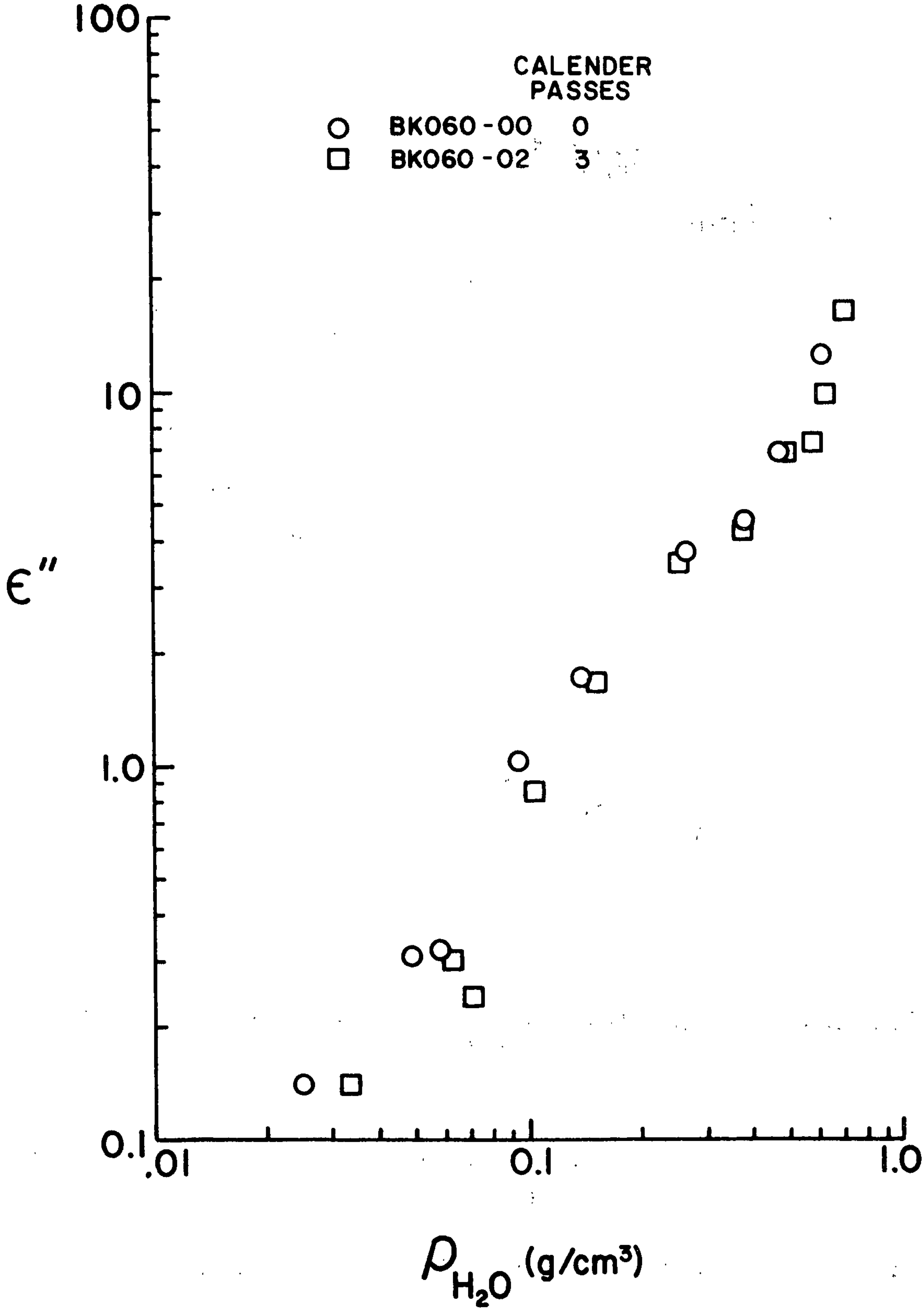

Figure 8. The Data of Figure 6 Replotted as log ε" versus log ρH_2O

If the curves in Fig. 7 and 8 are extrapolated to unit water density, the results are $\varepsilon \sim 50 + 25i$. This is higher than that of the unbleached kraft samples and is closely approaching the pure water value. In fact, the ε' curve for the BK samples is slightly above the UK curve over the range of water densities. We take these observations as evidence that the water molecules in the unbleached kraft samples are more strongly bound to fiber than in the bleached kraft. That is, they are not as free to oscillate and contribute to ε'. In addition, from Table II in PR1 it is noted that at the same relative humidity, the bleached kraft contains less moisture than the unbleached kraft, indicating that the unbleached kraft holds water more easily.

Figures 3, 4, 7, and 8 show that at high moistures, bleaching, wet pressing, refining, and calendering have only a small influence on the dielectric constant of paper, when compared to the overwhelming effect of changing water density. This explains why microwave moisture gages have been so successful in measuring high moistures in paper (and felts).

For machine-made papers, preferential alignment of the fibers in the machine direction results in a larger dielectric constant when the electric field is in the machine direction. This is discussed in PR1. To see if this effect persists at high moistures, a machine-made newsprint was tested. Samples were cut so that the electric field could be aligned with the machine direction and with the cross direction. Figures 9, 10, 11, and 12, show that small differences were also present at high moistures.

If the curves in Fig. 11 and 12 are extrapolated to unit water density, the result is $\varepsilon \sim 40 + 12i$. This is less than observed for the unbleached kraft, indicating that the water is more tightly bound in the newsprint.

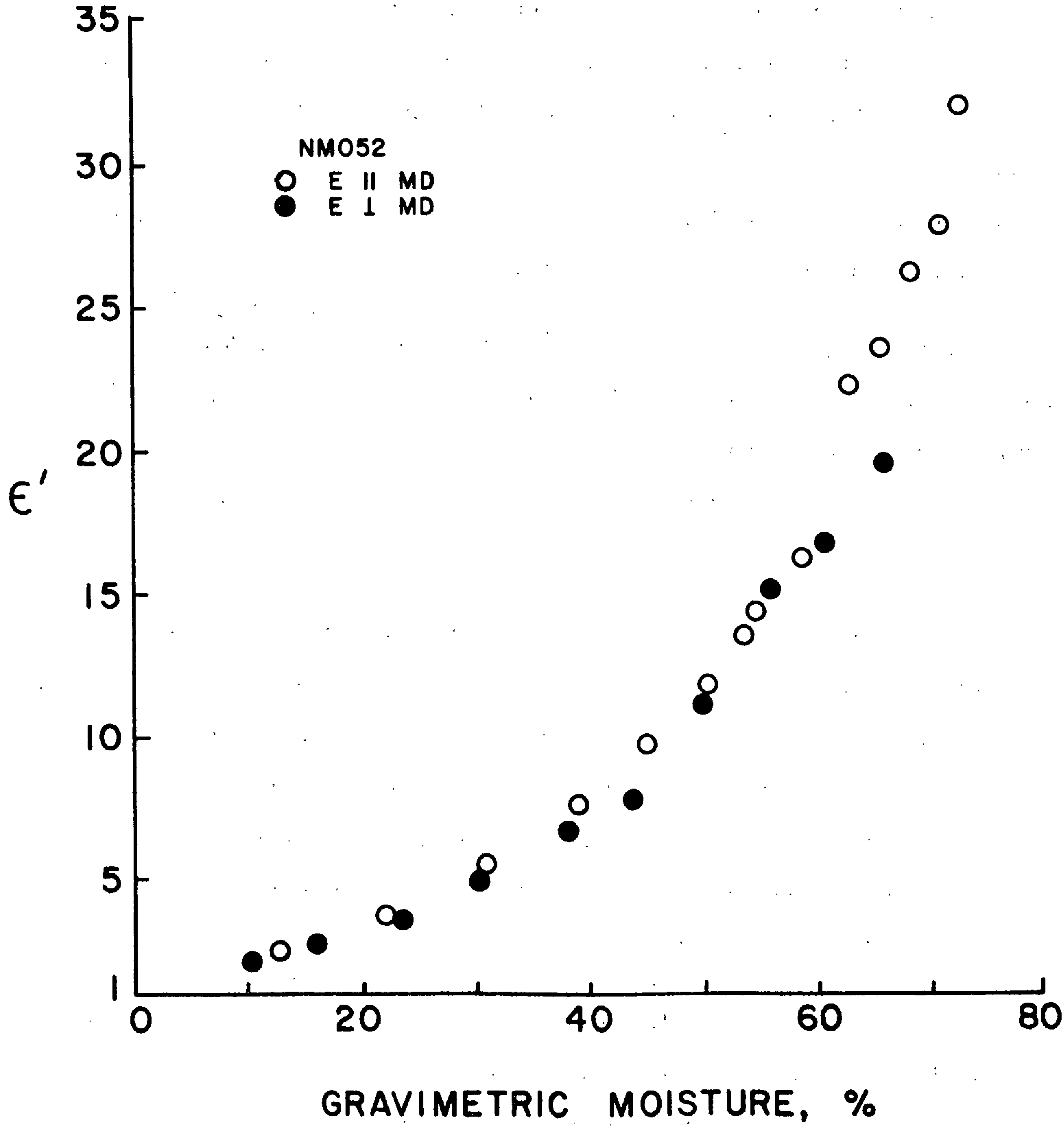

Figure 9. The Effect of Electric Field Orientation With Respect to the Machine Direction on the Real Part of the Dielectric Constant for a Newprint Sample. Measurements at 9.6 GHz

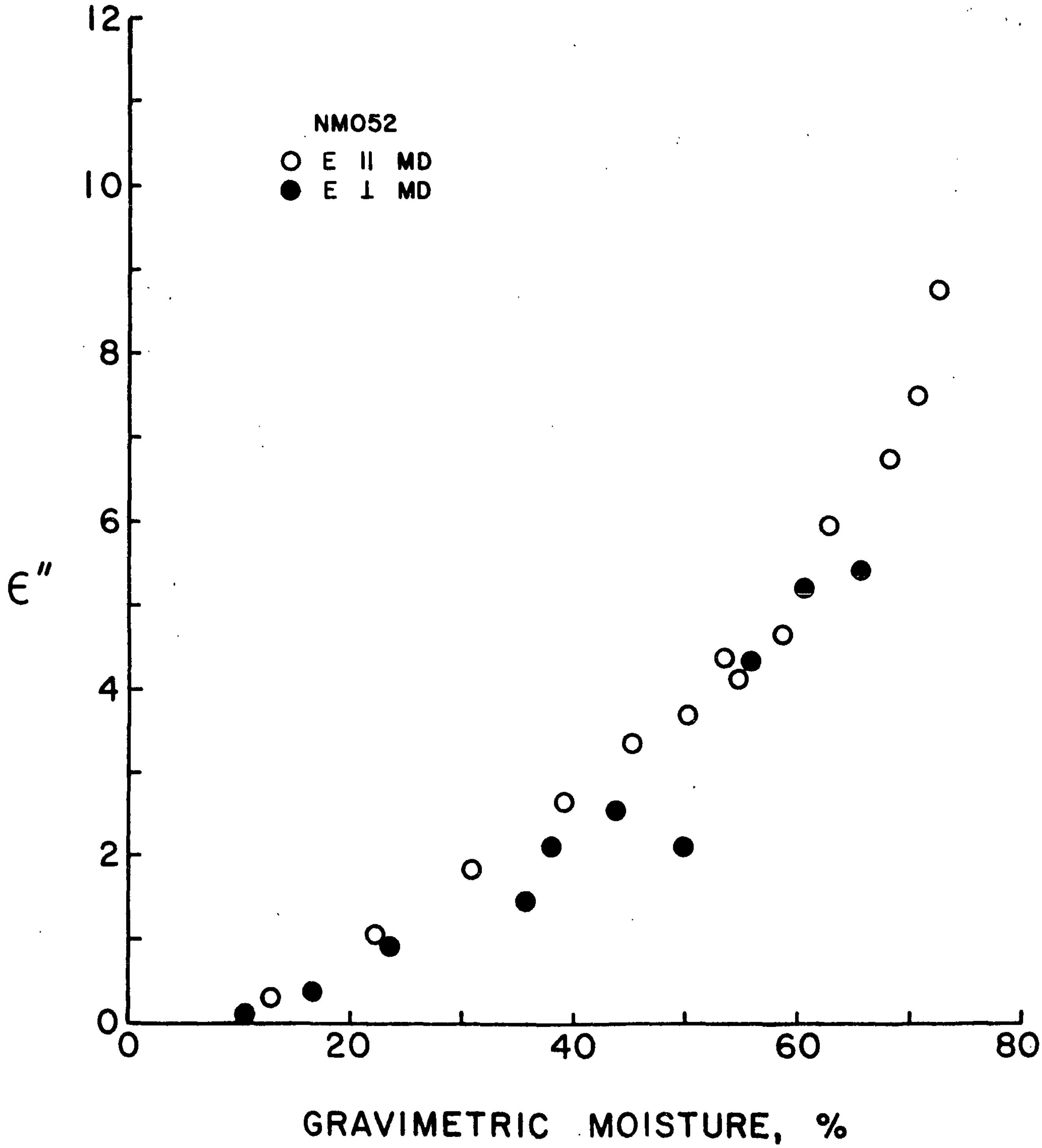

Figure 10. The Effect of Electric Field Orientation With Respect to the Machine Direction on the Imaginary Part of the Dielectric Constant for a Newsprint Sample. Measurements at 9.6 GHz

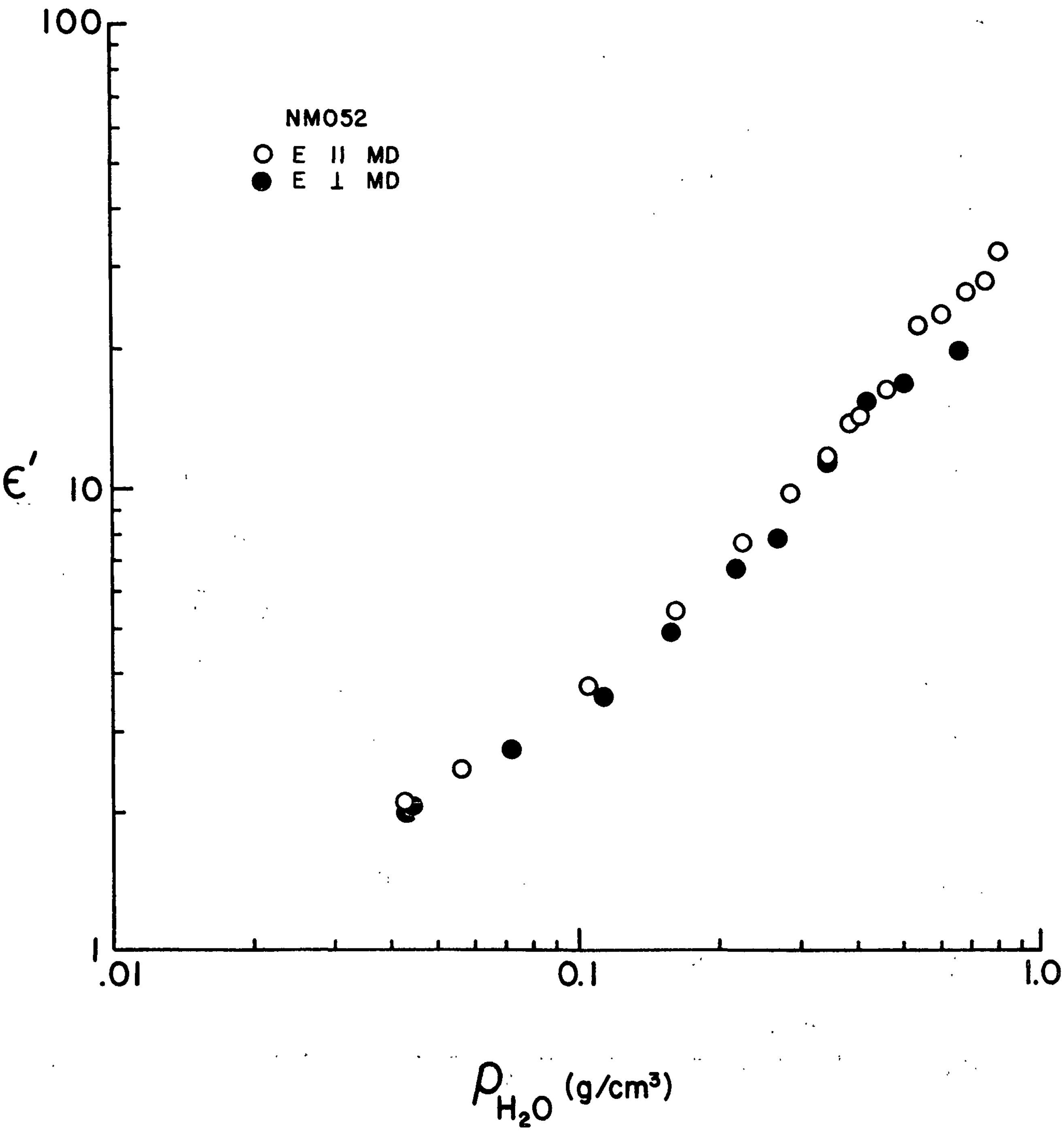

Figure 11. The Data of Figure 9 Replotted as log ε' <u>versus</u> log ρH_2O

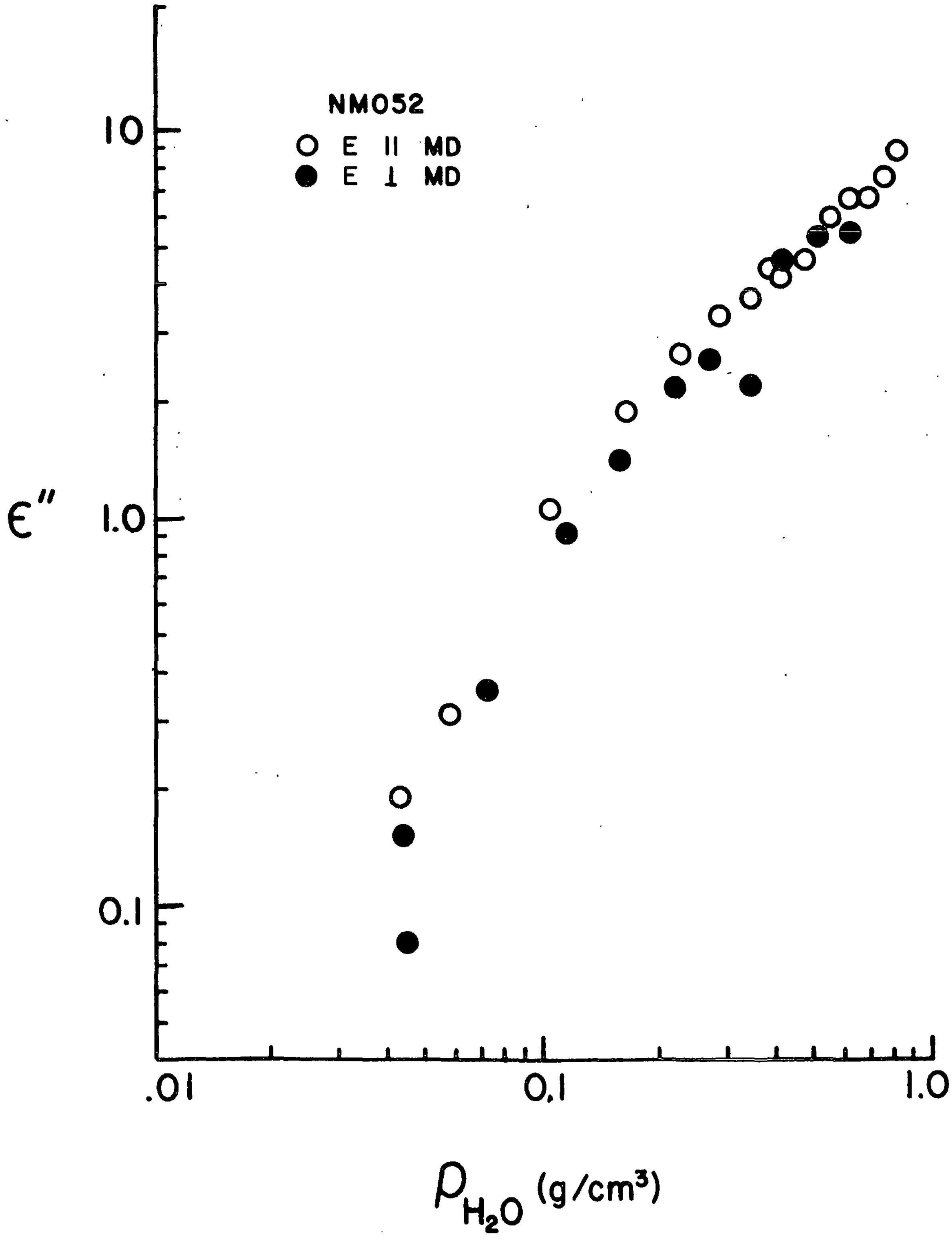

Figure 12. The Data of Figure 10 Replotted as log ε" <u>versus</u> log ρH_2O

FUTURE WORK

We are not planning any further activity in this area at this time. The data gathered on this project should be useful to designers of microwave moisture gages and driers. Several of the findings have special significance from a fundamental and practical standpoint. These are the anisotropy observed between the machine and cross machine directions, and the differences in water-fiber bonds inferred from the data.

If the anisotropy is only dependent upon fiber orientation, as proposed in PR1, then simultaneous measurements in these two directions on the moving paper web should give an indication of the fiber orientation in the web. Fiber orientation, in turn, is related to jet-wire speed ratios. At present, student research is directed toward understanding the nature of the dielectric anisotropy*.

The observation that the strength of water-fiber bonds affects the dielectric constant in a measureable way suggests that this or similar techniques might be used to further explore this bonding. Inasmuch as water in paper seriously affects all physical properties, an enhanced understanding of water-fiber bonds should be of considerable interest.

*Elmer Fleischman, Ph.D. Dissertation Research, "Mechanical and dielectric anisotropy in paper." The Institute of Paper Chemistry.

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